### **Supporting Information**

#### for

### The Influence of the Formal Replacement of Thiophenes by Stannoles in Terthiophene and Sexithiophene on the Optoelectronic Properties and Electrochemical Behavior

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### Chemicals

Table S1: List of used chemicals, their supplier, purity and further information.

Chemical	Supplier	Purity	Comments
Ammonium chloride	Carl Roth	> 99.5%	
2-Bromothiophene	TCI	> 98%	degassed by FPT, 3x
5,5'-Dibromo-2,2'-	ABCR	98%	
bithiophene			
Diphenyltin dichloride	VWR	>96%	Distilled prior use
Copper(I) chloride	Alfa Aesar	99.995%	stored in the glove box
Copper(I) iodide	Alfa Aesar	99.998%	stored in the glove box
2-Iodothiophene	TCI	> 98	
Magnesium sulfate	Grüssing	99%	
1,7-Octadiyne	<b>VWR</b> Chemicals	98%	degassed by FPT, 3x
[Pd(PPh <sub>3</sub> ) <sub>2</sub> Cl <sub>2</sub> ]	J&K	98%	stored in the glove box
[Pd(PPh <sub>3</sub> ) <sub>4</sub> ]	TCI	> 97	stored in the glove box
bis(Trimethylsilyl)acetylene	Sigma-Aldrich	99%	stored in the glove box freezer
Zirconium dichloride	ABCR	99%	stored in the glove box freezer

The reference for the spectroelectrochemical investigation, compound **StannoleOMe (13)**<sup>[1]</sup> was synthesized according to a literature procedure.

### Solvents

Table S2: List of solvents used and their purification.

Solvent	Supplier	Purity	Comments
Dichloromethane	VWR	HPLC	used as received
Cyclohexane	VWR	HPLC	used as received
Diethyl ether	VWR	99%	distilled prior use
N,N-Dimethylformamide	Acros Organics	99.8%	Extra dry
<i>n</i> -Hexane	VWR	HPLC	used as received
Methanol	<b>Fischer Scientific</b>	HPLC	used as received
2-Methyltetrahydrofuran	Fischer Scientific	HPLC	used as received
<i>n</i> -Pentane	VWR	tech.	distilled,
			dried via SPS, degassed by FPT (5x)
Pyridine	Acros Organics	99.8%	Extra dry
Toluene	VWR	HPLC	dried via SPS, degassed by FPT (5x)
Triethylamine	ChemPur	99%	anhydrous, degassed by sparging
			with argon

### Chromatography

Column chromatography was carried out by using the column machine PuriFlash 4250 from Interchim. Silica gel columns of the type PF (PuriFlash) 50 ( $\mu$ m grain size) SiHP (Silica gel High Performance) PF-50SiHP-JPF0120, and PF-50SiHP-JP-F0220 were used. The sample was applied using a dry load method using Celite<sup>®</sup>.

### **Synthesis**

1,8-Bis(thiophen-2-yl)octa-1,7-diyne (5)



The synthetic procedure was adapted from the literature.<sup>1</sup> In a glovebox, a mixture of 2-iodothiophene (3, 5.15 g, 24.5 mmol), [Pd(PPh<sub>3</sub>)<sub>4</sub>] (555 mg, 480 μmol) and copper(I)iodide (91.4 mg, 480 μmol) in TEA (8.0 mL) and DMF (14 mL) was stirred at 20 °C. After addition of 1,7-octadiyne (4, 1.30 g, 12.3 mmol), the red-brown suspension was stirred at 55 °C for 20 h. The reaction was quenched with a saturated solution of NH<sub>4</sub>Cl (1 x 30 mL). The aqueous layer was extracted with diethyl ether (3 x 30 mL) and the combined organic layers were washed with H<sub>2</sub>O (1 x 100 mL). The phases were separated and dried over MgSO<sub>4</sub>. After filtration, the volatiles were removed in vacuo. The residue was purified by column chromatography (silica gel, 1/20 dichloromethane/*n*-hexane,  $R_f = 0.18$ ) to give the product as a yellow waxy solid (5, 2.15 g, 7.95 mmol, 65%). <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.17 (dd, <sup>3</sup>J = 5.2 Hz, <sup>4</sup>J = 1.0 Hz, 2H, Tph-*H*-5), 7.12 (dd, <sup>3</sup>*J* = 3.6 Hz, <sup>4</sup>*J* = 1.0 Hz, 2H, Tph-*H*-3), 6.94 (dd, <sup>3</sup>*J* = 5.2 Hz, 3.6 Hz, 2H, Tph-*H*-4), 2.54 – 2.43 (m, 4H, C=C-CH<sub>2</sub>), 1.82 – 1.72 (m, 4H, C=C-(CH<sub>2</sub>)-CH<sub>2</sub>) ppm.<sup>13</sup>C{<sup>1</sup>H} NMR (150 MHz, CDCl<sub>3</sub>): δ = 131.1 (Tph-C-3), 126.9 (Tph-C-5), 126.1 (Tph-C-4), 124.1 (Tph-C-2), 94.0 (C=C-CH<sub>2</sub>), 74.2 (C=C-CH<sub>2</sub>), 27.9 (C=C-CH<sub>2</sub>), 19.4 C=C-(CH<sub>2</sub>)-CH<sub>2</sub>) ppm. HRMS (EI, 70 eV, MAT95, direct): *m/z* calcd. for C<sub>16</sub>H<sub>14</sub>S<sub>2</sub> [M-H]<sup>+</sup> 270.05314 found 270.05306. IR (ATR): υ = 3107 (w), 2939 (w), 2860 (w), 1516 (w), 1449 (w), 1427 (m), 1355 (w), 1294 (w), 1275 (w), 1237 (w), 1222 (w), 1185 (m), 1141 (w), 1083 (w), 1043 (w), 1015 (w), 897 (w), 846 (m), 829 (s), 741 (w), 691 (s), 662 (m) cm<sup>-1</sup>. Mp.: 44 °C

Rosenthal's reagent (6)



The synthetic procedure was adapted from the literature.<sup>2, 3</sup> In a glovebox, Cp<sub>2</sub>ZrCl<sub>2</sub> (5.00 g, 17.1 mmol) and *bis*(trimethylsilyl)acetylene (2.91 g, 17.1 mmol) were dissolved in THF (100 mL). At the Schlenk line, *n*-BuLi (2.5 M in hexanes, 13.7 mL, 34.2 mmol) was added dropwise at -78 °C over the course of 40 min. The yellow reaction mixture was stirred for 10 min. The cooling bath was removed and the reaction mixture was allowed to warm to 22 °C over the course of 2 h. The flask was transferred into a glovebox and pyridine (1.38 mL, 17.1 mmol) was added to the deep crimson solution, leading to a colour change to opaque purple. The solvent was removed *in vacuo* under inert conditions until ca. 1 mL remained. *n*-Pentane (250 mL) was added and the solution was stirred for 24 h. Then, the reaction mixture was filtered with PTFE syringe filters (0.45 µm pore size) and placed in a -30 °C freezer in a glovebox for 72 h. The supernatant was removed by decantation and the long black-purple crystals were dried *in vacuo* (**6**, 5.06 g, 10.8 mmol, 63%). <sup>1</sup>H NMR (500 MHz, C<sub>6</sub>D<sub>6</sub>):  $\delta$  = 8.85 (dt, <sup>3</sup>*J* = 4.9 Hz, <sup>4</sup>*J* = 1.7 Hz 2H, Py-H-2,6), 6.81 (tt, <sup>3</sup>*J* = 7.6 Hz, <sup>4</sup>*J* = 1.7 Hz, 1H, Py-H-4), 6.42 (ddd, <sup>3</sup>*J*<sub>HH</sub> = 7.6 Hz, 4.9 Hz, <sup>4</sup>*J* = 1.5 Hz, 2H, Py-H-3,5), 5.47 (s, 10H, Cp-H), 0.32 (s, 18H, CH<sub>3</sub>) ppm. <sup>13</sup>C{<sup>1</sup>H} NMR (126 MHz, C<sub>6</sub>D<sub>6</sub>):  $\delta$  153.9 (Py-C-2,6), 136.5 (Py-C-4), 123.1 (Py-C-3,5), 111.1 (*C*≡C), 106.4 (Cp-C), 2.5 (CH<sub>3</sub>) ppm.

2,2'-(2,2-Diphenyl-4,5,6,7-tetrahydro-2*H*-benzo[c]stannole-1,3-diyl)dithiophene (1)



The synthetic procedure was adapted from the literature.<sup>1</sup> In a glovebox, 1,8-bis(thiophen-2-yl)octa-1,7-diyne (7, 800 mg, 2.96 mmol) and Rosenthal's zirconocene (1.39 g, 2.96 mmol) were dissolved in toluene (8 mL). The dark red solution was stirred at 22 °C for 18 h under a N₂ atmosphere. Diphenyltin dichloride (1.02 g, 2.96 mmol) and copper(I)chloride (30 mg, 30 µmol) in toluene (2 mL) were added to the solution. The reaction mixture was stirred at 22 °C for further 6 h. The reaction was guenched with H<sub>2</sub>O (1 x 100 mL) and extracted with DCM (3 x 100 mL). The combined organic layers were dried over MgSO<sub>4</sub>, filtered and concentrated in vacuo. The crude product was purified by column chromatography (silica gel 15  $\mu$ m grain size, *n*-hexane, R<sub>f</sub> = 0.36) to give the product as a yellow solid (**1**, 540 mg, 1.01 mmol, 34%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.63 (dd, <sup>3</sup>J<sub>SnH</sub> = 22.3, 17.2 Hz, <sup>3</sup>J<sub>HH</sub> = 6.4 Hz, <sup>4</sup>J<sub>HH</sub> = 3.0 Hz, 4H, Ph-H-2,6), 7.41 – 7.32 (m, 6H, Ph-H-3,4,5), 7.27 – 7.25 (m, 2H, Tph-H-5), 6.99 – 6.91 (m, 4H, Tph-H-3,4), 2.94 – 2.86 (m, 4H, CH<sub>2</sub>), 1.85 – 1.78 (m, 4H, CH<sub>2</sub>-CH<sub>2</sub>) ppm.<sup>13</sup>C{<sup>1</sup>H} NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  = 149.8 (<sup>2</sup>J<sub>SnC</sub> = 38.6 Hz, stannole-C-3), 145.9 (<sup>2</sup>J<sub>SnC</sub> = 35.8 Hz, Tph-C-2), 138.3 (<sup>1</sup>J<sub>SnC</sub> = 248.4 Hz, Ph-C-1), 137.4 (<sup>2</sup>J<sub>SnC</sub> = 22.3 Hz, Ph-C-2,6), 130.2 (stannole-C-2), 129.5 (<sup>4</sup>J<sub>SnC</sub> = 6.5 Hz, Ph-C-4), 129.4 (<sup>3</sup>J<sub>SnC</sub> = 11.7 Hz, Tph-C-3), 129.0 (<sup>3</sup>J<sub>SnC</sub> = 27.6 Hz, Ph-C-3,5), 127.1 (Tph-C-5), 125.9 (Tph-C-4), 32.0 (CH<sub>2</sub>), 23.4 ((CH<sub>2</sub>)-CH<sub>2</sub>) ppm. <sup>119</sup>Sn{<sup>1</sup>H} NMR (187 MHz, CDCl<sub>3</sub>): δ -82.4 (s) ppm. HRMS (EI, 70 eV, MAT95, direct): *m/z* calcd. for C<sub>28</sub>H<sub>24</sub>S<sub>2</sub><sup>116</sup>Sn [M]<sup>+</sup> 540.03314 found 540.03303. IR (ATR): υ = 3060 (w), 2935 (w), 2359 (w), 1633 (w), 1515 (w), 1493 (w), 1478 (m), 1428 (m), 1411 (w), 1381 (w), 1348 (w), 1329 (w), 1283 (w), 1250 (w), 1211 (m), 1189 (w), 1134 (w), 1074 (m), 1059 (m), 1020 (w), 996 (w), 966 (w), 924 (w), 848 (m), 801 (m), 781 (w), 723 (m), 686 (s) cm<sup>-1</sup>. Mp.: 156 °C.

2-(Octa-1,7-diyn-1-yl)thiophene (9)



The synthetic procedure was added from the literature.<sup>4</sup> In a glovebox,  $[Pd(PPh_3)_2Cl_2]$  (52 mg, 74 µmol) and copper(I)iodide (35 mg, 67  $\mu$ mol) were added to a solution of 5,5'-dibromo-2,2'-bithiophene (10, 602 mg, 1.86 mmol) and 2-(octa-1,7-diyn-1-yl)thiophene (9, 700 mg, 3.72 mmol) in triethylamine (30 mL) and toluene (30 mL) at 25 °C. This heterogeneous yellow mixture was stirred for 2 d at 25 °C. Subsequently, all volatiles were removed in vacuo, and the residue was extracted with diethyl ether (3 x 100 mL) and all volatiles were removed again. After purification by column chromatography on silica gel (column machine, 120 g, cyclohexane/diethyl ether, 99:1), the product was isolated as yellowish solid (9, 715 mg, 1.32 mmol, 71%). <sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.18 (dd, <sup>3</sup>J = 5.2 Hz, <sup>4</sup>J = 1.2 Hz, 2H, Tph-*H*-3), 7.14 (dd,  ${}^{3}J$  = 3.6 Hz,  ${}^{4}J$  = 1.1 Hz, 2H, Tph-*H*-5), 7.01 (d,  ${}^{3}J$  = 3.8 Hz, 2H, biTph-*H*-4), 6.97 (d,  ${}^{3}J$ = 3.8 Hz, 2H, biTph-H-3), 6.94 (dd, <sup>3</sup>J = 5.2, 3.6 Hz, 2H, Tph-H-4), 2.50 (m<sub>c</sub>, 8H, C≡C-CH<sub>2</sub>), 1.70 (m<sub>c</sub>, 8H, C=C-CH<sub>2</sub>-CH<sub>2</sub>) ppm. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>): δ = 137.1 (biTph-C-5), 132.0 (biTph-C-3), 131.1 (Tph-C-3), 126.9 (Tph-C-4), 126.1 (Tph-C-5), 124.1 (Tph-C-2), 123.6 (biTph-C-4), 123.2 (biTph-C-2), 95.4 (Tph-C=C), 93.9 (biTph-C=C), 74.2/74.1 (Tph-C=C), 27.81/27.8 (C=C-CH<sub>2</sub>), 19.5/19.4 (C=C-CH<sub>2</sub>-CH<sub>2</sub>) ppm. HRMS (EI, 70 eV, MAT95, direct): *m/z* calculated for C<sub>32</sub>H<sub>26</sub>S<sub>4</sub> [M-H]<sup>+</sup> 538.09119 found 538.09200. IR (ATR): v = 3073 (w), 2936 (w), 2898 (w), 2861 (w), 2839 (w), 2220 (w), 1510 (w), 1423 (w), 1184 (w), 1044 (w), 865 (m), 845 (m), 829 (m), 694 (s) cm<sup>-1</sup>. Mp.: 91.5°C.

#### 5,5'-Bis(8-(thiophen-2-yl)octa-1,7-diyn-1-yl)-2,2'-bithiophene (11)



The synthetic procedure was added from the literature.<sup>4</sup> In a glovebox, [Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>] (52 mg, 74 µmol) and copper(I)iodide (35 mg, 67  $\mu$ mol) were added to a solution of 5,5'-dibromo-2,2'-bithiophene (10, 602 mg, 1.86 mmol) and 2-(octa-1,7-diyn-1-yl)thiophene (9, 700 mg, 3.72 mmol) in triethylamine (30 mL) and toluene (30 mL) at 25 °C. This heterogeneous yellow mixture was stirred for 2 d at 25 °C. Subsequently, all volatiles were removed in vacuo, and the residue was extracted with diethyl ether (3 x 100 mL) and all volatiles were removed again. After purification by column chromatography on silica gel (column machine, 120 g, cyclohexane/diethyl ether, 99:1), the product was isolated as yellowish solid (**11**, 715 mg, 1.32 mmol, 71%). <sup>1</sup>H NMR (601 MHz, CDCl<sub>3</sub>): δ = 7.18 (dd, <sup>3</sup>J = 5.2 Hz, <sup>4</sup>J = 1.2 Hz, 2H, Tph-H-3), 7.14 (dd, <sup>3</sup>J = 3.6 Hz, <sup>4</sup>J = 1.1 Hz, 2H, Tph-H-5), 7.01 (d, <sup>3</sup>J = 3.8 Hz, 2H, biTph-H-4), 6.97 (d, <sup>3</sup>J = 3.8 Hz, 2H, biTph-H-3), 6.94 (dd, <sup>3</sup>J = 5.2, 3.6 Hz, 2H, Tph-H-4), 2.50 (m<sub>c</sub>, 8H, C=C-CH<sub>2</sub>), 1.70 (m<sub>c</sub>, 8H, C=C-CH<sub>2</sub>-CH<sub>2</sub>) ppm. <sup>13</sup>C{<sup>1</sup>H} NMR (151 MHz, CDCl<sub>3</sub>): δ = 137.1 (biTph-C-5), 132.0 (biTph-C-3), 131.1 (Tph-C-3), 126.9 (Tph-C-4), 126.1 (Tph-C-5), 124.1 (Tph-C-2), 123.6 (biTph-C-4), 123.2 (biTph-C-2), 95.4 (Tph-C=C), 93.9 (biTph-C=C), 74.2/74.1 (Tph-C=C), 27.81/27.8 (C=C-CH<sub>2</sub>), 19.5/19.4 (C=C-CH<sub>2</sub>-CH<sub>2</sub>) ppm. HRMS (EI, 70 eV, MAT95, direct): *m/z* calculated for C<sub>32</sub>H<sub>26</sub>S<sub>4</sub> [M-H]<sup>+</sup> 538.09119 found 538.09200. IR (ATR): v = 3073 (w), 2936 (w), 2898 (w), 2861 (w), 2839 (w), 2220 (w), 1510 (w), 1423 (w), 1184 (w), 1044 (w), 865 (m), 845 (m), 829 (m), 694 (s) cm<sup>-1</sup>. Mp.: 91.5°C.

# NMR Spectra



Figure S2: <sup>13</sup>C{<sup>1</sup>H} NMR (600 MHz) spectrum of compound 1,8-bis(thiophen-2-yl)octa-1,7-diyne (5) in CDCl<sub>3</sub>.

220 210 200 190 180 170 160 150 140 130 120 110 100 90 ppm

80 70 60 50 40 30 20 10 0



Figure S4: <sup>13</sup>C{<sup>1</sup>H} NMR (126 MHz) spectrum of Rosenthal's zirconocene (6) in C<sub>6</sub>D<sub>6</sub>.

2,2'-(2,2-Diphenyl-4,5,6,7-tetrahydro-2*H*-benzo[c]stannole-1,3-diyl)dithiophene (1)



Figure S6: <sup>13</sup>C{<sup>1</sup>H} NMR (126 MHz) spectrum of compound 1 in CDCl<sub>3</sub>.



Figure S7: <sup>119</sup>Sn{<sup>1</sup>H} NMR (186 MHz) spectrum of compound 1 in CDCl<sub>3</sub>.



Figure S8: <sup>1</sup>H NMR (600 MHz) spectrum of 2-(octa-1,7-diyn-1-yl)thiophene (9) in CDCl<sub>3</sub>.



Figure S9:  ${}^{13}C{}^{1}H$  NMR (600 MHz) spectrum of 2-(octa-1,7-diyn-1-yl)thiophene (9) in CDCl<sub>3</sub>.

5,5'-Bis(8-(thiophen-2-yl)octa-1,7-diyn-1-yl)-2,2'-bithiophene (11)



Figure S10: <sup>1</sup>H NMR (600 MHz) spectrum of 5,5'-bis(8-(thiophen-2-yl)octa-1,7-diyn-1-yl)-2,2'-bithiophene (11) in CDCl<sub>3</sub>.





Dimer (2)



Figure S12: <sup>1</sup>H NMR (600 MHz) spectrum of compound 2 in CDCl<sub>3</sub>.



Figure S13:  $^{13}C\{^{1}H\}$  NMR (600 MHz) spectrum of compound 2 in CDCl3.



Figure S14: <sup>119</sup>Sn{<sup>1</sup>H} NMR (223 MHz) spectrum of compound 2 in CDCl<sub>3</sub>.

### Decomposition of the Dimer (2)



Figure S15: Solutions of stannole 1 (left) and 2 (right) in  $CDCl_3$  as prepared (a) and after 180 min (b) upon exposition to ambient light.

The respective <sup>1</sup>H NMR spectra of **2** showed unassignable signals in the aromatic region and a shift of the signals for the cyclohexyl protons to a higher field (Fig. S16).



**Figure S16**: <sup>1</sup>H NMR (600 MHz) spectra before (top) and after (bottom) exposition of **2** towards the light for 2 h in CDCl<sub>3</sub>. The signals at approx.  $\delta$  = 3.0 and 1.9 ppm are dedicated to the CH<sub>2</sub> protons of the cyclohexyl ring.



Figure S17: <sup>119</sup>Sn{<sup>1</sup>H} NMR (223 MHz) spectra before (top) and after (bottom) exposition of 2 towards light for 2 h in CDCl<sub>3</sub>.

## Thermogravimetric Analysis



**Figure S18**: Thermogravimetric analysis of stannole **1**, **2** and ter- and sexithiophene with a heating rate of 10 °C/ min under a nitrogen flow of 20 mL/min in an open aluminium crucible (40  $\mu$ L).

The rapid mass loss of terthiophene is probably due to sublimation processes.<sup>5</sup>

### Literature

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